Calibration Device for Reference Leak Value by Soap Film Flowmeter

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Abstract

The reference leak is divided into positive pressure reference leak and vacuum reference leak.In order to calibrate the leak rate of the positive pressure reference leak, a calibration device for reference leak value by soap film flowmeter was built, which can calibrate the positive pressure reference leak. The calibration device is composed of the automatic control part of the air source pressure, the measured reference leak and the standard soap film flowmeter. The device is used to calibrate reference leaks with a flow range from 1mL/min to 10L/min and a pressure range of positive pressure (1~1500) kPa. The relative expanded uncertainty of the device is 2.8%. A reference leak with a nominal pressure of 27.58 kPa and a nominal leak rate of 24.88 mL/min is selected for the test, and the uncertainty of the test result is evaluated. The relative expanded uncertainty of the calibration result of the leak $U_r(Q)$ is 2.9% (k=2). The value is more than 2.8%, which proves that it is reasonable to set the relative expanded uncertainty of this device at 2.8%.

1. Introduction

The reference leak is a calibrated leak with a known leak rate under specified conditions [1-2]. The reference leak allows the gas to flow from the inlet end of the throttling element to the outlet end according to the difference in pressure or concentration at both ends of the leak， so the leak rate is generated under the action of the throttling element.According to whether the outlet pressure is vacuum or atmospheric pressure [3-4]. It is divided into vacuum reference leak and positive pressure reference leak [5-7].

Positive pressure leak is a device that provides stable gas flow to atmospheric pressure which is often used to calibrate leak detectors working in sniffer mode and other leak rates under positive pressure conditions.The main calibration methods used are constant pressure method, constant volume method and dynamic comparison method, which can cover the calibration of positive pressure leak rate in the range of (10^{-3}~10^{-7}) Pa·m^3/s. Among them, the constant pressure method has the advantages of low measurement lower limit, high measurement efficiency, and small measurement uncertainty, and is the main research direction of various countries in the world. It is widely used in aerospace, air condition, electric power, microelectronics, semiconductor and other industries [8-9].

The vacuum leak is a device that provides stable gas flow to the vacuum end (outlet pressure less than 1 kPa) which is often used to calibrate mass spectrometer leak detectors. It is widely used in aerospace, electronic industry, electric power industry and refrigeration industry[10-11].

2. Calibration device

Chongqing Academy of Metrology and Quality Inspection has built a calibration device for reference leak value by soap film flowmeter, which can calibrate the positive pressure reference leak. The calibration device is composed of the automatic control part of the air source pressure, the measured reference leak and the standard soap film flowmeter. The device is used to calibrate reference leaks with a flow range from 1mL/min to 10L/min and a pressure range of positive pressure (1~1500) kPa. The relative expanded uncertainty of the device is 2.8 %. The physical diagram of the device is shown in Figure 1.

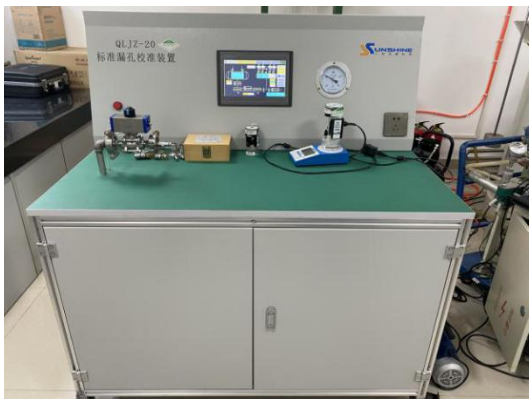

Figure 1: Physical diagram of the calibration device for positive pressure reference leak.

The overall structure of the device is shown in Figure 2.The gas source enters the volume buffer tank 1 (not more than 20 L) after passing through the pressure reducing valve. The volume tank is used to balance and stabilize the temperature of the gas source, and the pressure is stable at about (1.6~1.7) MPa. The specific value of pressure is monitored by a precision digital pressure transmitter. The gas in the volume buffer tank 1 is adjusted by the electro-pneumatic proportional pressure valve F1 of (70~1500) kPa, and then divided into two channels to enter the subsequent process.

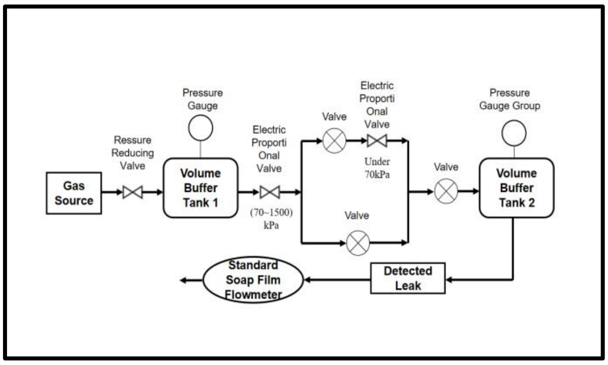

Figure 2: Overall structure of the calibration device for positive pressure reference leak.

The gas in one channel passes through the the electro-pneumatic proportional pressure valve F1, then enters into the volume buffer tank 2 directly. This channel can carry out calibration work with the reference leak in pressure of (70~1500) kPa.

In the other channel, the gas passes through the electro-pneumatic proportional pressure valve F1firstly. Then passes through electro-pneumatic proportional pressure valve F2 of (0~70) kPa which adjusts the gas pressure to (1~70) kPa. After entering into the buffer tank 2, it can carry out calibration work with the reference leak below pressure of 70 kPa.

After the air flow reaches the desired calibrated pressure, the pressure is stabilized again through volume buffer tank 2 (no more than 2 L). At the same time, a standard group of precision digital pressure transmitters (3 units, 0.1 level) are set on the pressure stabilization tank to provide pressure information. Then the air flow passes through the tested reference leak and the standard soap film flowmeter successively for calibration.

The whole device adopts PLC (as shown in Figure 3) control mode to realize automatic pressure adjustment and automatic measurement of the system, and the pressure of gas source remains stable in the range of flow (leak rate) from 1mL/min to 10 L/min.

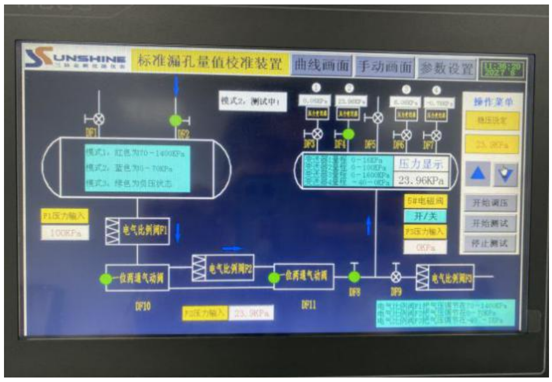

Figure 3: PCL automatic control diagram.

3. Test data

Select a reference leak (the nominal pressure of the leak is 27.58kPa, and the nominal leak rate is 24.88mL/min) to conduct the test on this device. During the test, the gas source pressure of the device is stable at 27.58kPa. The measured results is shown in Table 1.

Table 1: A set of measurement results of the reference leak calibration device

Measurement Times	Measured Leak Rate Value (mL/min)
1	24.37
2	24.02
3	23.88
4	23.74
5	24.78
6	25.16
7	24.86
8	25.33
9	23.21
10	24.78
Measured Leak Rate Value (Average Value, mL/min)	24.41

4. Uncertainty evaluation of measurement results[12-13]

4.1 Measurement model

$$Q = \overline{Q} \times k \tag{1}$$

in the formula:
Q-- the measured leak rate, mL/min;
$\overline{Q}$-- the average of measured leak rate, mL/min;
k--coefficient (usually 1) due to the calibration method or conditions.

4.2 Relative standard uncertainty $u_r(Q)$

$$u_r(Q) = \sqrt{u_r^2(\overline{Q}) + u_r^2(k)} \tag{2}$$

in the formula:
$u_r(Q)$-- relative standard uncertainty of the leak rate measurement results;
$u_r(\bar{Q})$-- relative standard uncertainty introduced by measurement repeatability;
$u_r(k)$-- relative standard uncertainty of the coefficient due to the calibration method or conditions.

4.3 Relative standard uncertainty $u_r(k)$ of coefficient due to calibration method or conditions

The coefficient mainly includes factors such as soap film flowmeter, temperature fluctuation, pressure fluctuation of the front gas source, pressure measurement and atmospheric pressure fluctuation, etc. It is expressed by the following formula:

$$k = k_1 \times k_T \times k_{p1} \times k_{p2} \times k_{p3} \tag{3}$$

in the formula:
k_1-- the influence of soap film flowmeter on the measurement results;
k_T-- the influence of temperature fluctuations on the measurement results;
k_{p1}-- the influence of the pressure fluctuation of the front gas source on the measurement results;
k_{p2}-- the influence of the previous pressure measurement error of the on the measurement results;
k_{p3}-- the influence of atmospheric pressure fluctuations on the measurement results.

Among them, the influencing factors are independent and uncorrelated with each other. $u_r(k)$ is calculated by the following formula:

$$u_r(k) = \sqrt{u_r^2(k_1) + u_r^2(k_T) + u_r^2(k_{p1}) + u_r^2(k_{p2}) + u_r^2(k_{p3})} \tag{4}$$

in the formula:
$u_r(k_1)$--relative standard uncertainty introduced by soap film flowmeter;
$u_r(k_T)$-- relative standard uncertainty introduced by temperature fluctuation;
$u_r(k_{p1})$-- relative standard uncertainty introduced by the pressure fluctuation of the front gas source;
$u_r(k_{p2})$-- relative standard uncertainty introduced by pressure measurement;
$u_r(k_{p3})$-- relative standard uncertainty introduced by atmospheric pressure fluctuation.

4.4 Relative standard uncertainty $u_r(\bar{Q})$ introduced by measurement repeatability

In this test, the reference leak was measured 10 times with the calibration device, and the arithmetic mean value was taken as the calibration result. The measurement repeatability was expressed by the experimental standard deviation. The Bessel formula is used to find the standard deviation, and it is calculated by the following formula:

$$S(x) = \sqrt{\frac{\sum_{i=1}^{10}(x_i - \bar{x})}{10-1}} = 0.68 \tag{5}$$

$$u_r(\bar{Q}) = \frac{S_x}{\sqrt{10} \times 24.41} \times 100\% = 0.89\% \tag{6}$$

in the formula:
$U_r(\bar{Q})$-- relative standard uncertainty introduced by measurement repeatability;
$S(x)$-- the repeatability of the calibrated leak rate, mL/min;
$\bar{x}$ -- The average value of 10 measured leak rate values, mL/min。

4.5 Relative standard uncertainty introduced by soap film flowmeter $u_r(k_1)$

It is directly introduced according to the grade requirements of the electronic soap film flowmeter in the calibration device.The relative expanded uncertainty of the electronic soap film flowmeter is 1% (k=2)， so the relative standard uncertainty introduced by this item is:

$$u_r(k_1) = \frac{1\%}{2} = 0.5\% \tag{7}$$

4.6 Relative standard uncertainty introduced by temperature fluctuation $u_r(k_T)$

Because the single measurement period is short, the relative standard uncertainty component introduced by temperature fluctuation is small and can be ignored.

4.7 Relative standard uncertainty introduced by the pressure fluctuation of the front gas source $u_r(k_{p1})$

Because the influence of the pressure fluctuation of the front gas source on the measurement results needs to be calculated by different calculation formulas according to the specific structure of each leak , the calculation is relatively complicated. To simplify the calculation, the relative standard uncertainty introduced by this term is:

$$u_r(k_{p1}) \approx 1\% \tag{8}$$

4.8 Relative standard uncertainty introduced by pressure measurement $u_r(k_{p2})$

Calculate the pressure measurement error introduced by pressure gauges with different accuracy levels, and use this to calculate the relative standard uncertainty. The smaller the pressure value, the larger the relative standard uncertainty is introduced under the same measuring range. The grade of the pressure gauge of this calibration device is 0.1, and the nominal pressure of this leak to be tested is 27.58 kPa. When the pressure gauge of (0~100) kPa measures the gas source pressure of 27.58 kPa, it is assumed that the measurement error obeys a uniform distribution. The relative standard uncertainty introduced by the term is:

$$u_r(k_2) = \frac{0.1\% \times 100}{\sqrt{3} \times 27.58} \times 100\% = 0.21\% \quad (9)$$

*4.9 R*elative standard uncertainty introduced by atmospheric pressure fluctuation $u_r(k_{p3})$

Because the single measurement period is short, the relative standard uncertainty component introduced by the fluctuation of atmospheric pressure is small and can be ignored.

4.10 Relative standard uncertainty of the leak rate measurement results $u_r(Q)$

According to the calculation of the above formula, the measurement repeatability of the reference leak is 0.68 mL/min, and the relative uncertainty introduced by the repeatability is 0.89%. The measurement of the leak is actually the assignment of the leak, once the mechanical structure of the leak is fixed, the uncertainty of the measurement result mainly comes from the reference leak calibration device. The relative standard uncertainty caused by temperature fluctuation and atmospheric pressure fluctuation are much smaller than the relative standard uncertainty caused by repeatability and calibration device, so they can be ignored in uncertainty evaluation.

The main factors that affect the uncertainty of the calibration result include measurement repeatability, soap film flowmeter, pressure fluctuation of the front gas source, pressure measurement, etc. Through uncertainty analysis, the relative standard uncertainty components of the leak rate calibration results are shown in Table 2.

Table 2: Relative standard uncertainty components of the leak rate.

Sources of Ancertainty	Symbol	Value/%
Measurement Repeatability	$u_r(\bar{Q})$	0.89
Soap Film Flowmeter	$u_r(k_1)$	0.5
Front Source Pressure Fluctuation	$u_r(k_{p1})$	1
Pressure Measurement	$u_r(k_{p2})$	0.21

The components are independent and uncorrelated with each other, so the relative composite standard uncertainty is:

$$u_r(Q) = \sqrt{u_r^2(\overline{Q}) + u_r^2(k_1) + u_r^2(k_{p1}) + u_r^2(k_{p2})} \quad (10)$$

$$u_r(Q) = \sqrt{(0.89\%)^2 + (0.5\%)^2 + (1\%)^2 + (0.21\%)^2} \approx 1.44\% \quad (11)$$

$$U_r(Q) = 2u_r(Q) = 2.88\% \approx 2.9\% \quad (12)$$

The leak rate of this leak is 24.41 mL/min. The final relative expanded uncertainty of the calibration result $U_r(Q)$ is 2.9% (k=2) by calculating the combined standard uncertainty and relative expanded uncertainty.

5. Conclusion

This positive pressure leak calibration device can calibrate the reference leaks with the gas source pressure range of (1~1500) kPa and the leak rate of 1mL/min~10L/min. The relative expanded uncertainty of the device is 2.8%.

The whole device adopts PLC control mode to realize automatic pressure adjustment and automatic measurement of the system, so as to provide a stable air source pressure.

In this paper, a reference leak with a nominal pressure of 27.58 kPa and a nominal leak rate of 24.88 mL/min is selected for the test, and the uncertainty of the test result is evaluated. The relative expanded uncertainty of the calibration result of the leak $U_r(Q)$ is 2.9% (k=2). The value is more than 2.8%, which proves that it is reasonable to set the relative expanded uncertainty of this device at 2.8%.

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